RESEARCH ARTICLE

Structural Diversity in Organotin(IV) Carboxylates: A Study of X-Ray Crystallography

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Abstract: Among the organotin compounds, organotin(IV) carboxylates adapt a fascinating range of structural diversity, including monomer, dimer, tetramer, polymer, oligomer ladder, cyclic and hexameric drums structures. Generally four- and five coordinated complexes are observed in triorganotin(IV) complexes. While diorganotin(IV) complexes displayed a large range of structures, including five, six and seven coordinate geometries with distorted trigonalbipyramidal, octahedral and pentagonal bipyramidal structures. Distannoxanes complexes are also observed in diorganotin(IV) complexes. A comprehensive review, organotin(IV) carboxylates are presented with particular reference to their structural studies as described bycrystallography methods.

Keywords: Organotin(IV) Carboxylate, Structural Chemistry, X-Ray crystallography

Introduction

The organotin(IV) carboxylates have been known due to its versatile and significant important biological activities¹⁻¹⁰. A number of studies have been done on organotin(IV) carboxylates by various spectroscopic studies and the molecular structure of these complexes have described by x-ray crystallography^{11,12}. Organotin(IV) carboxylates have been known due to its notable and vast structural diversity^{8,9,13-16}. Interesting tin chemistry has been observed in organotin(IV) carboxylate with different coordination sphere at tin atom. Generally five- and six coordinated organotin(IV) complexes are observed with trigonalbipyramidal and octahedral geometry, respectively. Four- and seven coordinated geometry have also been observed in tricyclohexyltin¹⁷ and trinuclear di-*n*-butyltin(IV) complexes¹⁸, respectively. Yip *et al.*,¹⁹ and A. Rehman *et al.*,²⁰ have also synthesized four-coordinatedtriorganotin(IV), complexes bis[triphenyltin(IV)] succinate and (3-amino-4-chlorobenzoato)trimethyltin(IV), respectively, with monoclinic crystal system and monodentate bonding of the carboxylate group to the tin atom.

Amini *et al.*, have synthesized bis(benzoato) ethyl phenyltin(IV) by the reaction of ethyldiphenyltin(IV) iodide with silver benzoate in ethanol, by the cleavage of a phenyl

group bound to tin and suggested a skew-trapezoidal bipyramidal structure. A cyclic hexamer structure of triorganotin carboxylates, ethyldiphenyltin(IV) acetate and diphenylpropyltin(IV) acetate has also been determined by them²¹. Trimethyltin(IV) 2,3-methylenedioxy benzoate has been synthesized by Hussain *et al.* It has been found that the structure is tetrahedral for triorganotin(IV) complex, in solution state. While in the solid state, triorganotin(IV) complex showed the distorted trigonal bipyramidal geometry²². The di- and triorganotin(IV) complexes with *n*-butylhydrogen phthalate have been synthesized by the reaction of corresponding organotin(IV) oxide with *n*-butylhydrogen phthalate in dry toluene. A distorted octahedral geometry was found for diorganotin(IV) derivatives and triorganotin(IV) *n*-butylphthalates adopt linear, polymeric trigonalbipyramidal geometry in which *n*-butylphthalate is a monoanionicbidentate coordinating through the C(O)O group²³.

The coordination sphere at tin atom also plays a significant role in their important biological activities. Win et al., have synthesized organodistannoxane dimer complexes derived from alkylaminobenzoic acids. In these complexes carboxylate anions acted as monoand bidentate ligand²⁴. These complexes have been found active against human liver carcinoma cells (HepG2), in which bis[2-(methylamino)benzoato] tetrabutyldistannoxane(IV) dimer showed significant activity. Sun et al.,²⁵ have synthesized polynuclear phenyl- and butyltin(IV) carboxylates having ladder and drum structures and showed anti-tumor activities against HepG2 human hepatocellular liver carcinoma cells, A549 human lung carcinoma cells and B16-F10 melanoma cells. Among these complexes a centrosymmetricphenyltin(IV) complex exhibited the best cytotoxicity. A trimethyltin(IV) complex derived from 2,6dimethoxypyridine-3-carboxylic acid has also displayed significant anti-tumor activities against human adenocarcinoma HeLa, human myelogenous leukaemia K562, human malignant melanoma Fem-x and human breast carcinoma MDA-MB-361²⁶. Shukla et al.,²⁷ have investigated significant antitumor, antibacterial, antifungal and insecticidal activity of some triorganotin(IV) carboxylates. Organotin(IV) complexes with *n*-butylhydrogen phthalate²⁸ and 2-(N-Maleoylamino)-2-methylpropanoate²⁹ also displayed remarkable antibacterial and antifungal activity against various pathogenic strains. Keeping in mind a vast structural diversity of organotin(IV) carboxylates, an attempt is being made in this article to present a brief up to date review on the organotin(IV) carboxylates with special reference to their structural chemistryas determined by crystallographicmethods.

Structural chemistry of organotin(IV) carboxylates

In organotin(IV) carboxylates, carboxylate group of ligand coordinates to the tin atom in monodentate or bidentate fashion in different stochiometries and leads to a different coordination sphere at tin atom. This section will be discuss as seven-, six-, five-, four-coordinated complexes and distannoxanes.

Seven coordinated complexes

In organotin complexes, distorted pentagonal bipyramidal geometry results by asymmetrical coordination of the carboxylate ligands to the Sn atom. A representative structure is given in Figure 1. A centrosymmetric dimer is observed in the complex, $\{[Ph_2Sn(2,6-C_5H_3N)(COO)][Na(2,6C_5H_3N)(COOH)(COO)(CH_3OH)_2]\}$. The coordination sphere of each tin atom is N₁O₄C₂ in a distorted pentagonal bipyramidal, with two phenyl groups are trans to each other (C-Sn-C angle of 167.52(8)°) (Figure 1). Each tin atom is coordinated by two monodentate and one bidentate carboxylate group³⁰. Salam *et al.*,¹⁸ have also synthesized a seven coordinated tri-nuclear di-*n*-butyltin(IV) complex of pyruvic acid-*N*(4)- cyclohexyl-

thiosemicarbazone in distorted pentagonal bipyramidal geometry. The central tin(IV) atoms are bonded to carboxylato-O, the azomethine-N and the thiolato-S atoms of ligand and bridges between the tin atoms are formed through the carbonyl oxygen atom of the carboxylate groups. They have reported that crystal of this complex is cubic with space group $I-4_3d$, a = b = c = 30.3273(17) Å, $\alpha = \beta = \gamma = 90^\circ$, Z = 16 and final $R_1 = 0.0390$, $wR_2 = 0.0843$ for observed reflections $4582(I > 2\sigma(I))$.



Figure 1. Molecular structure of complex { $[Ph_2Sn(2,6C_5H_3N)(COO)][Na(2,6C_5H_3N)(COOH)(COO)(CH_3OH)_2]$ }

Six coordinated complexes

A distorted octahedral geometry is found in six coordinated complexes as shown in Molecular structure of complex $[Me_2Sn(2,6-C_5H_3N)(COO)_2(H_2O)]$.H₂O (Figure 2). The crystal and molecular structure of $[Me_2Sn(2,6-C_5H_3N)(COO)_2(H_2O)]$.H₂O has been determined by A. Azadmeher et al.,³⁰. The tin atom, bonding to two methyl carbons (Sn1-C8 2.092(5), Sn1-C9 2.085(5) Å; C9-Sn1-C8 164.8(2)°), two carboxylate oxygen atoms (Sn1-O1 2.435(3), Sn1-O2 2.182(3) Å; O2-Sn1-O1 139.14(11)°), pyridine nitrogen (Sn1-N1 2.287(4) Å) and a water molecule (Sn1-O1W 2.300(3) Å) has a distorted octahedral geometry (Figure 2).



Figure 2. Molecular structure of complex [Me₂Sn(2, 6-C₅H₃N)(COO)₂(H₂O)].H₂O

Amini *et al.*,²¹ have reported that crystal of ethylphenyltin(IV) dibenzoate PhEtSn[OC(O)C₆H₅]₂ is triclinic with space group *P1* (a=8.6426(18), b= 10.472(2), c= 11.520(2) Å; α = 83.594(16)°, β = 76.512(16)°, σ =87.126(17)°; Z= 2, D_{calc}(g cm⁻³) = 1.540, *R_{int}*= 3935 (0.021)). A skew-trapezoidal bipyramidal geometry, with the two organic groups in the pseudo-axial positions towards the weaker equatorial Sn-O interactions is observed in the complex (Figure 3). A skew-trapezoidal planar geometry is also observed in a Di-*n*-butyltin(IV) bis- [2,3-bis (4-chlorophenyl)propenoato-O,O] complex³¹.



Figure 3. Molecular structure of complex PhEtSn[OC(O)C₆H₅]₂

Baul *et al.*,³² have studied x-ray crystal structure of triphenyltin(IV) complex derived from carboxylate ligand, potassium β -{[(E)-1-(2hydroxyphenyl)methylidene]amino}-propionate. A one-dimensional polymeric structure is observed with two Sn-atoms with distinct coordination environments. The SnPh₃ moieties are linked by two modes of bridging alternating along the polymeric chain (Figure 4).



Figure 4. The unique repeat unit in the polymeric chain structure of $[Ph_3SnHL]_n$ (50% probability ellipsoids)

The coordination sphere of Sn(1) is C_3O_3 , with three phenyl groups, a carboxylate *O*-atom from one carboxylate ligand and the phenoxide *O*-atom from a second carboxylate ligand, and second carboxylate *O*-atom of former carboxylate ligand, which coordinates strongly to Sn(2), also has a weak interaction with Sn(1) (Sn...O= 3.202(3) Å) and completing six coordination at Sn(1). While the same coordination sphere of Sn(2) is built

with three phenyl groups, a carboxylate *O*-atom from one carboxylate ligand and two carboxylate *O*-atoms from a second carboxylate ligand (Sn(2)-O(2)=2.396(3), Sn(2)-O(31)=2.148(3), Sn(2)...O(32)=2.986(3) Å).

Five coordinated complexes

Among the organotin(IV) complexes, triorganic complexes form a polymeric structure with trigonal bipyramidal geometry^{22,32,33}. Monomer tructures are also observed in tribenzyltin(IV) derivatives^{34,35}. A trigonal bipyramidal geometry is also observed in dimethyltin(IV) complex, {Me₂Sn[OC(O)(C₁₄H₉)]₂.CH₃OH} CH₃OH with two carboxylates at axial and equatorial positions and the coordination number is raised to five as the methanol occupies the apical position of the trigonal bipyramid³³. A cyclic hexamer structure is observed in ethyldiphenyltin- (Figure 5) and diphenylpropyltin(IV) acetate (Figure 6), with the electronegative oxygen atoms occupying the apical positions and the phenyl and ethyl or propyl groups lying in the equatorial plane. The Sn-O bond lengths are observed in the ranges from 2.103 Å to 2.394 Å for ethyldiphenyltin derivative and 2.200 Å to 2.314 Å, indicating the asymmetric bonding in each tin centre²¹.



Figure 5. Molecular structure of complex ethyldiphenyltin acetate [EtPh₂SnOC(O)CH₃] Ellipsoids represent 30% probability levels



Figure 6. Molecular structure of complex diphenylpropyltin(IV) acetate [Ph2PrSnOC(O)CH₃]. Ellipsoids represent 50% probability levels

The geometry at the Sn atom is suggested as intermediate between square pyramidal and cis-trigonal bipyramidal in triphenyl meclofenamic complex, $[Ph_3Sn[(mef)]_3$ described by Demertzi *et al.*,⁶. In this complex carboxylate group acts as an anisobidentate chelating ligand (Sn₂...O (1) 2.079(3) Å, Sn... O(2) 2.629(4) Å) and spans equatorial and axial sites. A 1D helical chain (Figure 7) and 1D polymeric chain structure (Figure 8) is described in triphenyltin- and trimethyltin(IV) derivative of 6-chloro-3-pyridineacetic acid respectively, with a trigonal bipyramidal geometry at each tin centre.



Figure 7. The 1D spring-like helical chain structure of complex $\{(Ph_3Sn)[(COO) (CH_2C_5H_3NCl)]\}_n$ (the phenyl groups are omitted for clarity)



Figure 8. The 1D infinite zigzag chain structure of complex $\{(Me_3Sn)[(COO) (CH_2C_5H_3NCl)]\}_n$

In complex {(Ph₃Sn)[(COO)(CH₂C₅H₃NCl)]}_n the coordinated oxygen atoms occupy the axial sites (Sn-O= 2.18(3)–2.32(4) Å) and the O-Sn-O angles at 173.8(15)–179.3(15)°, indicating the presence of a linear arrangements³⁴. A monomeric trans R₃SnO structural moiety (Figure 9) is suggested in tribenzyltin(IV) complex, Bz₃Sn[O₂CC₆H₄{N=N(C₆H₃-4-OH(C(H)=NC₆H₄Cl-4))}-o](H₂O).

In this complex, tin atom has a distorted trigonal-bipyramidal geometry with equatorial benzyl groups $(Sn-C(21)=2.150(2) \text{ Å}, Sn-C(28)=2.148(2) \text{ Å}, Sn-C(35)=2.143(2) \text{ Å}; C(21)-Sn-C(28)=118.19(9)^\circ, C(21)-Sn-C(35)=116.35(9)^\circ, C(28)-Sn-C(35)=123.18(9)^\circ)$ and the axial positions occupied by an O atom from the carboxylate ligand $(Sn-O(1)=2.150(1) \text{ Å}; O(1)-Sn-C(21)=89.12(8)^\circ, O(1)-Sn-C(28)=96.40(8)^\circ, O(1)-Sn-C(35)=99.12(7))^\circ$ and the O atom from the water ligand $(Sn-O(4)=2.399(2) \text{ Å}; O(1)-Sn-O(4) 173.58(6)^\circ)$. The carbonyl O atom of the carboxylate ligand also coordinates very weakly to the Sn atom with long Sn...O(2) distances of $3.056(2) \text{ Å}^{35}$.



Figure 9. The molecular structure of $Bz_3Sn[O_2CC_6H_4{N=N(C_6H_3-4-OH(C(H)=NC_6H_4Cl-4))}-o](H_2O)$ showing the atom-labelling scheme (50% probability ellipsoids)

The molecular structure of trimethyltin(IV) 2,3-methylenedioxy benzoate²² and tributyl{ β -{[(*E*)-1-(2hydroxyphenyl) methylidene]amino} ropionate} tin(IV)³² are shown to be a polymeric chain structure having Monoclinic crystal system with space group *P*2₁/*c* (a= 10.7071(7), b= 9.9808(10), c= 12.4504(8) Å; $\alpha = \gamma = 90$, $\beta = 111.976(7)^{\circ}$; Z= 4; D_c (g cm⁻³)= 1.771) (Figure 10) and *P*2₁/*n* (a= 13.7830(3), b= 9.9060(2), c= 17.7972(4) Å; $\alpha = \gamma = 90$, $\beta = 109.616(1)^{\circ}$; Z=4; D_c (g cm⁻³)= 1.399) (Figure 11), respectively. The geometry of the tin is described as a trigonal bipyramidal geometry with oxygen atoms of carboxylate group in the apical positions (Sn1-O1= 2.376(3), Sn1-O2= 2.217(3) Å) and the 3 methyl groups in the equatorial positions (C11-Sn1= 2.122(5), C10-Sn1 2.122(6), C9-Sn1 2.128(5) Å) in aforementioned complex. While in tributyl{ β -{[(*E*)-1-(2hydroxyphenyl)methylidene] amino} ropionate} tin(IV), the coordination sphere of the Sn-atom is trans- -Bu₃SnO₂ in trigonal bipyramidal geometry with three butyl groups at equatorial position (Sn-C(11)= 2.147(2), Sn-C(15)= 2.151(2), Sn-C(19)= 2.140(2) Å) and two carboxylate O atoms at axial positions (Sn-O(1)= 2.460(2), Sn-O(2)ⁱ= 2.181(2), Sn...O(1)ⁱ= 3.043(2) Å).



Figure 10. An ORTEP¹⁸ drawing of complex trimethyltin(IV) 2,3-methylenedioxy benzoate. Displacement ellipsoids are drawn at 50% probability level



Figure 11. A three-unit segment of the polymeric tributyl{ β -{[(*E*)-1-(2hydroxyphenyl) methylidene]amino}propionato} tin(IV) (50% probability ellipsoids)

Tian *et al.*,³⁶ have studied X-ray crystal structure of organotindinuclear complex, (HL)SnPh₃•Ph₂SnL [L=2-O-3,5-Br₂C₆H₃CH=NCH(*i*-Pr)COO] derived from triphenyltin chloride and potassium N-[(3,5-dibromo-2-hydroxyl-phenyl)methylene] valinate in benzene. The coordination spheres of tin atoms are trans- O₂Sn1C₂N and trans- O₂Sn2C₃ in distorted pentagonal bipyramidal geometry (Figure 12).



Figure 12. The molecular structure of (HL)SnPh₃•Ph₂SnL

In O_2Sn1C_2N coordination sphere, two phenyl groups and imino N1 atom occupied the equatorial positions and a phenoxide O1 and a carboxylate oxygen O2 atom occupied the axial positions (O1-Sn1-O2= $156.92(16)^{\circ}$) and while three phenyl groups occupied the equatorial positions around Sn2 atom and the carbonyl oxygen O3 atom of bridging carboxylate group and the carboxylate group oxygen O4 atom of the other ligand occupied the axial positions (O3-Sn2-O4 178.35(16)°).

Four coordinated complexes

A four coordinated complexes are observed with the monodentate bonding of the carboxylate group to the tin atom in distorted tetrahedral geometry. Yip *et al.*,¹⁹ have synthesized Bis[triphenyltin(IV)] succinate by reacting triphenyltin(IV) hydroxide with substituting succinic acid and observed a four coordinated complex with monoclinic crystal system having space group $P2_1/n$ and a = 9.5257(7), b = 19.2278(14), c = 9.7482(7) Å; $\alpha = \gamma = 90$, $\beta = 102.9070(10)^0$, Z =4 (Figure 13).



Figure 13. *ORTEP* plot for complex bis[triphenyltin(IV)] succinate at 50% probability level with atom numbering scheme

A four coordinated trimethyltin(IV) complex, (3-amino-4-chlorobenzoato)trimethyltin (IV) is also synthesized by Rehman *et al.*,²⁰ in which three methyl groups (Sn-C= 2.118 (2)-2.119 (2) Å) and one O atom (Sn-O= 2.0804 (12) Å) is bonded to the tin atom in distorted tetrahedral geometry (Figure 14). The complex is crystallize in monoclinic crystal system with space group $P2_{1/2}c$ and a = 11.9077 (7), b = 9.1237 (5), c = 12.6554 (7) Å; β = 113.086 (1)⁰, Z =4. Furtheremore the crystal structure of this complex contains centrosymmetric dimmers formed via intermolecular N-H....O hydrogen bonds (Figure 15).



Figure 14. The molecular structure of (3-amino-4-chlorobenzoato)trimethyltin(IV) with displacement ellipsoids drawn at the50% probability level. H atoms are shown as spheres of arbitrary radii



Figure 15. Centrosymmetric immers of (3-amino-4-chlorobenzoato)trimethyltin(IV), formed through intermolecular hydrogen bonding (dashed lines). H atoms not involved in hydrogen bonding have been omitted for clarity

Distannoxanes

In diorganotin(IV) complexes, distantion are observed with five or six coordinated geometry^{6,37-40}. A centrosymmetric dimer of an oxoditin(IV) complex, tetrabutylbis-(3,4-dichlorophenylacetato)distantion with monodentate and bridging bidentate carboxylate ligands is described by M.A. Saeed *et al.* The central Sn_2O_2 core is fused with 2 sixmembered rings ($Sn_2O_3C_1$) having O and C atoms of the ligand that show different modes of coordination with Sn (Figure 16).



Figure 16. The ORTEP-POV molecular structure of complex, tetrabutylbis(3,4dichlorophenylacetato)distannoxane drawn at the 30% probability level. For clarity, only the connecting C atoms of the *n*-butyl groups are shown

The coordination sphere of endocyclic and exocyclic Sn atoms are C_2SnO_3 in distorted trigonal-bipyramidal geometry with Sn-C distances lying in the range of 2.089(11)-2.181(14) Å, while Sn-O distances are in the range of 2.054(6)-2.257(9) Å⁴¹.



Figure 17. Molecular structure of tribenzyl{2-{[(2Z)-3-hydroxy-1methyl-2-butenylidene]amino}acetato}tin(IV)

A centrosymmetric dimer is also observed in tribenzyl{2-{[(2Z)-3-hydroxy-1methyl-2-butenylidene]amino}acetato}tin(IV) with a trigonalbipyramidal configuration at each tin atom (Figure 17). The three benzyl groups occupied the equatorial positions (Sn-C(21)= 2.1583(15), Sn-C(31)= 2.1610(15), Sn-C(11)= 2.1805(17) Å) while the axial positions are occupied by a carboxylate oxygen from one ligand molecule O(2) (Sn-O(2)#1= 2.2166(11) Å)

and the alkoxy oxygen, O(1) (Sn-O(1)= 2.3712(11) Å), of the second ligand molecule⁴². A ladder-like structure is found in a centrosymmetric dimer distannoxane, [Me₂(Meclo) SnOSn(Meclo)Me₂]₂ complex due to the fusion of a central Sn₂O₂ core with a outer Sn₂O₂ rings (Figure 18). The exocyclic Sn atom is six coordinated and shows distorted octahedral geometry. The endocyclic Sn atom is five coordinated and described as intermediate between square pyramidal and cis-trigonal bipyramida¹⁶. A drum structure is observed in [PhSn(O) (COO)(CH₂C₅H₃NC1)]₆ complex with a Sn₆O₆ central stannoxane core which contains two puckered six-membered [Sn₃(µ₃-O)₃] rings as its top and bottom faces and the side face of the drum is characterized by six puckered four-membered [Sn₂(µ₃-O)₂] rings³⁴.



Figure 18. Molecular structure of complex [Me₂(Meclo)SnOSn(Meclo)Me₂]₂

Baul *et al.*,³² have investigated the crystal structure of dibutyl{ β -{[(*E*)-1-(2-hydroxyphenyl)ethylidene]- amino} ropionate}tin(IV) (Figure 19) and dibutyl { β -{[(*E*)-1-(2-hydroxy-3 methylphenyl)ethylidene]amino} ropionate}tin(IV) (Figure 20) and found a centrosymmetric tetranuclear complexes containing a planar Sn₄O₂ core in which two μ_3 -oxo *O*-atoms connect an Sn₂O₂ ring to two exocyclic Sn-atoms. Both the complexes have six coordinations at each tin atom and crystallize in the triclinic crystal system with space group *P*1 and a= 12.475(1), b= 13.574(1), c= 14.212(1) Å; α = 94.149(5), β = 113.754(5), γ = 109.696(5)°; Z=1 for fore mentioned complex and a= 13.0368(3), b= 13.6627(3), c= 14.7318(3) Å; α = 113.936(1), β = 102.200(1), γ = 108.279(1)°; Z= 1 for dibutyl { β -{[(*E*)-1-(2-hydroxy-3 methylphenyl)ethylidene]amino} ropionate}tin(IV).



Figure 19. Molecular structure of complex dibutyl{ β -{[(*E*)-1-(2-hydroxyphenyl)-ethylidene]- amino} ropionate}tin(IV) (50% probabilityellipsoids)



Figure 20. Molecular structure of complex dibutyl { β -{[(*E*)-1-(2-hydroxy-3 methylphenyl)-ethylidene]amino}propionato}tin(IV) (50% probability ellipsoids)

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